Shale Oil Production in Estonia

Schieferölgewinnung in Estland
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Keltser

Oil shale is the most important mineral deposit of the Estonian SSR. The minable area has been estimated at 2,470 square kilometers and reserves at roughly 3,500 million tons. Present industrial methods of extracting oil from the shale result in an oil yield of 20 percent, which means that if the entire acount of s shale reserves could be converted to oil, 700 million tons would be produced.

The shale deposit has six minable layers which in the Kiviõli Mine are 2.7 meters thick. The shale yield is 1.7-2 tons per square meter, excluding layer A which, because of its high clay content, is, at present not being exploited at Kiviõli. The organic matter and ash content vary considerably, depending on the layer and location. The average for Kiviõli may be assumed to be as follows:

Organic matter	36 %
Mineral ash	46 %
CO_2	18 %
	100 %

Shale is mined both in open pits and in underground mines. The total output for 1936 was 765,000 tons and might reach one million tons in 1937. Of this 600,000 tons will be distilled and the rest used for fuel.

I. General

a. Preparation of the Shale

Preparation of the shale starts in the mine where the limestone is carefully removed from between the layers. When the shale comes from the mine, it still has a limestone content of 7-10 percent where the limestone has actually intruded into the layers.

The shale, coming from the mine, is first crushed to a maximum size of 100 millimeters then, with the aid of screens, it is sorted into three classes: fine shale - up to 10 millimeters in size; nut shale - from 10 to 25 millimeters in size; lump shale - from 25 to 100 millimeters in size.

The two latter varieties can be distilled without further processing, but the fine shale must first be made into briquettes of nodules of at least the size of nut shale.

b. Drying

In a certain sense drying, as employed in some plants, belongs to the preparation process. Large lumps of shale have a moisture content of from 12 to 16 percent when they come from the mine and the fine shale has a moisture content of 14 to 18 percent. If the shale is predried, the fine shale adhering to it can be more easily screened off and this is very important for the distillation process. Drum driers, formerly used to predry the shale, proved unsatisfactory since the pieces of shale break up in them. At present large belt driers are used.

c. Behavior of the Shale during Distillation

The behavior of shale during the thermal process presented a number of difficulties to the constructors of distillation furnaces:

When the shale is heated to 200 degrees Centigrade, its color darkens and the first signs of the formation of bitumen are noted. At 350 degrees Centigrade, the shale begins to soften. At 360 degrees Centigrade, oil starts to be formed and reaches a maximum point at 400-450 degrees Centigrade. From 450 to 480 degrees Centigrade oil formation slows down and, at 480 degrees the distillation is practically ended since only insignificant amounts of oil and gas are formed at a higher temperatures.

From 420 to 440 degrees Centigrade the process goes on exothermically.

The temperature range of from 360 to 410 degrees is dangerous since the shale is inclined to melt at this temperature and melting makes distillation exceedingly difficult or hinders it altogether. Melting is favored by a slow heating. Therefore it is of primary importance to ensure that the rate of heat application up to the formation of tar does not fall below a certain level.

II. Distillation Furnace Construction

In Estonia three different types of distillation furnaces are in use with all existing methods of introduction of heat to the charge:

1. Direct introduction of heat by passing a current of heating gas through the charge.

2. Indirect introduction of heat through the walls of the retort in which the charge has been placed and no direct contact of the heating gas with the charge.

3. Direct introduction of heat by a gas current employing the distillation vapors themselves as the heat transmitter rather than the heating gas.

The Pintsch generators, representative of the first group, have been in operation since 1925 in the State Shale Plant in Kokhtla.

The Davidson rotating retort, in operation since 1931 in the plant belonging to the New Consolidated Gold Fields Ltd, belongs to the second group.

The Estonian tunnel furnace of the Eesti Kivi͠oli A-Ü (Estonian Petroleum Co.) and the Gröndal-Zeidler-Carlson movable-grate furnace of the Estonian Oil Shale Consortium, operative since 1927, are representative of the third group.

a. The Pintsch Distillation Furnace of the State Shale Plant

The plant has 14 generators of the Pintsch type, six of which were put into operation in 1925 and eight in 1936. The apparatus, originally delivered by the Pintsch Firm, has been improved by technicians of the state plant and adapted to the peculiarities of shale. The generators with an original daily capacity of 33 tons now handle 35 to 37 tons daily.

Oil production is effected as follows:

Lump shale, 15 to 170 millimeters in size, previously crushed and freed of fine shale is delivered by bucket lift to the supply bunker. Here, after further screening to eliminate remnants of fine shale, it goes in an electrically-driven charge car which takes four to five 350-to 400-kilogram charges per furnace per hour to the feeding devices of the generator.

The generator itself is a 13-millimeter thick, riveted tin-plate cylinder, 6.15 meters high, with an external diameter of 2.70 meters, and lined with firebrick. Under the feeding device in the generator is a distribution cone. About 2.20 meters above the lower edge of the generator there are six pipe inlets through which cold gas can be blown into the generator. There are poke holes in the ceiling of the generator as well as half way up the side of it. The generator has a rotating grate without a water receptacle.

In the distillation area of the generator the shale sinks slowly and is warmed gradually. Temperatures of from 360 to 500 degrees prevail here, and the temperature is regulated in the dangerous temperature range by bringing back the cold gases which are separated off from the condensation installation.

The hot residue which has been subjected to a heat of 500-600 degrees Centigrade moves on to the combustion chamber where it is burned on a rotating grate, and the hot gases necessary for the distillation process are produced.

The oil vapors formed in the distillation area combine with the water vapor and the hot gases in the gas collector where a part of the heavy oil spearates and flows off. Part of the heavy oil is pumped back into the gas collector's spraying device to be used to eliminate some of the dust from the oil vapors. From the gas collector the mixture of vapor and hot gas goes to the air cooler, a hollow, cylindrical piece of apparatus, and from there to the preliminary cooler in which the greater part of the oil vapors are condensed. The mixture goes on to the secondary cooler where it is cooled to 25 degrees Centrigrade and the light oils separate off. Then it is washed with crude oil in the gasoline washers. The mixture leaving the gasoline washers, containing about 20 grams per cubic meter of light gasolines and carbohydrates, is, in part, brought back to the boiler combustion area, in part returned as cold gas to regulate the temperature in the distillation area, and in part blown off into the air.

The oil-water mixtures obtained in the condensation process are gathered in water-separating devices, heated to 60 degrees Centigrade, to facilitate the separation of the water, and then the oil, later to be used as a dust remover, is pumped into a tank. The gasoline-containing oil coming from the gasoline washers likewise goes to the water separators where it is warmed, neutralized with diluted sodium hydroxide, and, after removal of the water, pumped into the supply tank.

The oil yield in the distillation generators is 17.1 to 17.3 percent of the run-of-the-mine shale or 41 to 41.5 percent of the organic substance, that is about 68 to 70 percent of the yield in the Fischer apparatus. The amount of permanent gases escaping is about 644 cubic meters per ton of shale or about 25 times the permanent distilled gas.

The advantages of the generators lie in their low investment costs, their simple construction and maintenance, as well as in the use of distillation coke as a heating material. On the other hand the distillation generator is not completely adapted to the characteristics of the shale.

The great dilution of the distillation vapors makes the obtaining of gasoline very difficult so that today over 70 percent of the gasoline is lost with the waste gases. In the present installation only up to 8 grams per cubic meter of gasoline is washed out of gas containing up to 28 grams per cubic meters, that is, 20 grams per cubic meter are lost with the waste gases. Even if the installation of more efficient gasoline washers reduced this figure to 10 grams per cubic meter, 6 percent of the gasoline as referred to the shale would still be lost.

As a result of these phenomena the oil yield in reference to the amount of shale delivered is 2.5 to 3.5 percent lower than in the case of other types of furnace.

b. The Davidson Rotating Retort

The Davidson Rotating Retort is a distillation furnace for the indirect introduction of heat by external heating. The eight presently operating rotating furnaces are steel cylinders 23 meters long and with a diameter of 1.22 meters. The cylinder of the furnace is slightly inclined in the direction in which the shale moves forward. It is set on rollers and rotates once every minute and a half. It is powered by an electric motor set up at the entrance end of the cylinder. Inside the cylinder, scrapers move longitudinally back and forth in the distillation zone to prevent the shale and coke from adhering to the walls.

There is a continuous and automatic delivery from the feed hoppers of shale crushed up to 40 millimeters in size with a fine shale content of 30-40 percent.

The shale is moved gradually forward through the slope of the incline and is at the same time increasingly heated until at the exit it reaches a heat of 485 degrees Centigrade. The coke falls at the lower end into a hopper, the upper and lower surfaces of which are sliding surfaces moved by compressed air which make it possible to regulate the feeding of coke to the combustion chamber. This coke and the permanent gas recovered from the condensation area furnish the heat necessary for the distillation.

The oil vapors, created during the distillation, are drawn off by a gas pipe outlet, conducted through a dust catcher and go then to a condensation installation consisting of cooler pipes, washers, and a deep cooling installation.

The capacity of the retort is 20 to 21 tons daily. Experiments with a larger retort with a capacity of 75 tons per day have failed. Rich shale with an organic matter content of over 42 percent presents difficulties in distillation which can be overcome by thinning out the charge with limestone. The oil yield is given as 20-22 percent depending on the quality of the shale (referred to the yield in a Fischer apparatus this is equivalent to a yield of 88 percent).

There is relatively high dust content in the crude oil and the crude gasoline content in the crude oil amounts to 22-25 percent.

The possibility of utilizing considerable amounts of fine shale along with lump shale in the Davidson retort and the utilization of the residues of distillation for the creation of heat necessary for distillation are some of the advantages of this distillation furnace.

However, the samll capacity of the retort as well as the relatively high cost of the installation as referred to ton of daily yield make difficult the industrial utilization of this distillation furnace.

c. The Gröndal-Zeidler-Carlson Movable Grate Distillation Furnace

The Gröndal-Zeidler-Carlson Distillation Furnace was constructed by the Estonian Oil Shale Consortium in 1927/1928 and was put into operation in the spring of 1929. After a stoppage of 7 years, it was again put into operation in 1936. The distillation furnace is a tunnel furnace about 50 meters long, with entrance and exit chambers each 11 meters long. These can be separated from one another and from the outside air by slides. Tracks run through the furnace. There is room for 20 cars in the distillation area and for four each in the entrance and exit chambers. When a car is piled 300 millimeters in height it holds 1,500 kilograms of shale.

The heating is effected through two separate heating chambers arranged under the cars. The heating chambers have a rectangular cross section, are riveted out of heavy tin plate and connected with one another by compensators. (Kompensator) In each heating chamber there are vertical pipes with an inner diameter of 95 millimeters through which the distillation vapors circulate. The heating gases pass through the heating chambers and flow about the outside of the pipes. The total heating surface of the heating chambers amounts to 1,600 square meters.

The furnace has an outer casing of welded tin plate and is insulated against outside air. It has a capacity of 250 tons per day.

The oil yield is given as 19 to 22.7 percent referred to run-of-the mine shale, and the average can be taken at 20 percent. Referred to the Fischer apparatus, the yield for a working period of 17 days is given as 97.4 percent.

The furnace is suitable to the peculiar characteristics of shale in its distillation process and gives good results. However shutting off the outside air from the distillation area by a simple sliding device does not completely achieve the desired goal and the shale is somewhat dried in the distillation area.

d. The Estonian Tunnel Furnace

The Estonian tunnel furnace was developed by the Eesti Kiviõli A-Ü on the basis of years of experiments and operational experiences. In 1927/1928 a furnace with a daily capacity of 75 tons was constructed and gave such encouraging results that in 1930/1931, two furnaces were set up with a daily capacity of 250 tons each, and in 1936/1937, two more with a daily capacity of 400 tons each. In increasing the furnace. There is room for 20 cars in the distillation area and for four each in the entrance and exit chambers. When a car is piled 300 millimeters in height it holds 1,500 kilograms of shale.

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Three lock chambers divide the tunnel of the distillation furnace into three parts, the drying chamber, the distillation chamber, and the quenching chamber.

The furnace cars loaded with lump and nut shale are moved by hydraulic devices into the first lock which insulates the drying chamber from the outside air and then they go into the drying chamber where the shale is dried by superheated steam. The steam necessary for the drying is formed from the mine moisture content of the shale which had arrived from preliminary drying in the drying chamber with a moisture content of 6.5-9 percent. This steam is superheated by pipe superheaters which are heated from without by departing hot gases. Blowers bring about the circulation of the steam. The number of blowers corresponds to the number of cars in the drying chamber. Every 7-14 minutes the train is moved hydraulically forward by one car. The car moves into the second lock and from there into the distillation chamber. Here the volume of circulating gases and vapors is kept constant by drawing off by pipe the excessive amounts formed during distillation and moving them to the condensation area where liquid hydrocarbons are obtained in fractions.

After the car has passed through all parts of the distillation chamber, it enters the third lock and from there it goes to the quenching chamber. Here the residue from the distillation is sprayed with water and cooled. The car is emptied and the residue of distillation taken by cable car to the dump heap. The car is then moved back to the opening of the furnace, refilled and the cycle starts over again.

The 400-ton furnaces put out by the Eesti Kiviõli A.-Ü since 1937 have a tunnel with a diameter of 2 meters and 53 meters long. The furnaces have two drying places, 12 distillation places, and two quenching places and the second lock is equipped to serve as an additional distillation place. The tunnel body is divided into sections about 2.5 meters long connected with one another by compensators (Kompensator). The pipes connecting the superheater to the tunnel are likewise provided with compensators.

The distillation cars with a load piled 900 millimeters high carry 2.20 tons. The furnace handles about 400 tons per day. This corresponds to about 7.5 cars per minute [sic, must be hour] with an 8-minute stop at each distillation place before moving forward. With 13 distillation places, the distillation is carried on for 104 minutes which is adequate for the complete distillation of the shale.

The total heating surface of the 16-tube superheater is 472 square meters. The pipes of the distillation chamber's superheater are of alloyed, tinder and corrosion-proof steel with the quality varying according to the operating temperature of the pipe. In the case of the first four or five pipes very high quality is essential. The others may be made of ordinary iron pipes.

The oil yield averages 20 percent, ranging from 19.5 to 21.9 percent as referred to run-of-the-mine shale. In this connection it must be noted that relatively poor shale is distilled with a 34 to 36 percent organic matter content, which in the laboratory shows a yield of 64.8 percent referred to the organic matter. Referred to the yield in a Fischer apparatus the operational yield ranges from 94 to 99.8 percent and averages 96 percent.

The Estonian tunnel furnace fulfills all the demands of distillation, affords extensive adaptation to the peculiarities of the substance being distilled, and is suitable for the distillation of even intensely caking, oil-bearing rock.

2. Fractionated Condensation

The oil vapors leaving the distillation chamber at 470 degrees Centigrade first pass a preliminary cooler in which they are cooled down to 300 degrees Centigrade by being sprayed with hot heavy oil. From the preliminary coolers they go to the condensation installation which works as follows: The distillation vapors enter the air cooler at 300 degrees Centigrade and leave it at 230 degrees Centigrade. Here the heavy oil fraction with a specific gravity of 1.020 to 1.050 is separated out and after passing by a secondary cooler flows off into a mixing tank. From the air cooler the oil vapors enter the waterpipe cooler which they leave at 150 degrees Centigrade and during this stage of the process the medium oil fraction with a specific gravity of about 0.990 separates out. The medium oil passes a secondary cooler and, after water has been removed from it in the water separator, it likewise flows through the secondary cooler into the mixing tank. The mixture,of the two fractions, after the removal of moisture, goes to the tank for mixed oil.

From cooler No 1 the oil vapors proceed through coolers Nos 2, 3, and 4, leaving cooler No 2 at 60 degrees Centigrade, cooler No 3 at 25 degrees Centigrade, and cooler No 4 at 15 degrees Centigrade. In cooler No 2 light oil with a specific gravity of about 0.850 separates out and is conducted through a water separator to the light oil tank. The light oil fraction contains 45 to 50 percent gasoline which is separated off in a topping installation. In coolers Nos 3 and 4 gasoline with a specific gravity of about 0.760 is separated out, and is conducted to the gasoline tank via a water separator. The gas leaving cooler No 4 still contains about 450 grams of gasoline per cubic meter and is conducted to the deep cooling installation.

Fractionated condensation therefore results in gasoline-free oils, crude gasoline, and the gasoline-bearing light oil fraction which accounts for about 18 percent of the total amount of oil. This light oil has to be distilled to obtain the gasoline fraction.

3. The Deep Cooling Installation

The gas is drawn by a blower from the condensation installation to the deep cooling installation where the gasoline is, for the most part, freed by the aid of a calcium chloride solution and a gasoline wash. The deep cooling installation was first introduced by the Eesti Kivioli A.-Ü with the setting up of the 75-ton furnace. As a result of enlarging the installation, it has at present a capacity of 4,500 tons per year. The other oil shale plants have since set up such installations or are in the process of doing so.

An ammonia compressor installation with a capacity of 210,000 calories at minus 20 degrees Centigrade ammonia-evaporation temperature serves as the cold generator and makes it possible to cool the cooling fluids to minus 15 degrees Centigrade.

The deep cooling, or other installation for obtaining gasoline from gas, works as follows:

The deep cooling proceeds in two stages to which a third stage is added: the topping of the gasoline wash.

In the first stage a calcium chloride solution serves as the cooling fluid. This operates in two columns sprinkling the gas approaching it from the condensation unit. The gasolines condensed here flow first into settling units and then into the gasoline tank. The solution flows into a solution tank and begins the cycle anew by being pumped through an ammonia evaporator, cooled there and brought back to the columns. The gas which has been treated by the solution still contains 100 to 120 grams of gasoline per cubic meter and moves on through three washing columns D_1 to D_3, in a countercurrent, to the gasoline wash which has been cooled to minus 10 or minus 12 degrees Centigrade.

The gas that is not condensible in the washing columns, after passing column D_3, goes into the gas supply system leading to the combustion area of the furnaces.

The gasoline wash, containing gasoline-bearing gas is pumped through the heat exchanger, the gasoline wash installations, and deep coolers K_1 to K_3 back into the washing columns. The gasoline vapors escaping from the topper are condensed in water cooler K_1 and deep cooler TK_4. The uncondensed gasolines are brought to the gas supply system and begin the cooling cycle again.

The permanent gas returning to the combustion area of the furnace (averaging 25 cubic meters per ton of distilled shale) contains an average of only 10 grams of gasoline per cubic meter of gas, therefore 0.125 percent of the total oil yield or 0.025 percent as referred to the shale,- insignificant amounts.

III Conclusion

Distillation products are, them, crude oil and gasoline. The gasoline is ready to be marketed after the refining process, and many ways of further processing the crude oil are available. The chief products of the Estonian distillation industry are at present wood preservation oil, floor oil, fuel oil, bituminous asphalts and roof tars. Many problems still await solution, including the utilization of phenol, the manufacture of fuels, and lubricating greases.

8. Inside View of Condensation Installation Page 1147 of text

9. Diagram of Condensation Installation Page 1147 of text

Air cooler

Cooler 4 Cooler 3 Cooler 2 Cooler 1

Blower

Water separator of coolers 3 and 4

Water separator of cooler 2

Water separator of cooler 1

Secondary cooler 2

Mixture tank

Secondary cooler 1

Gas Pressure Control

Gasoline tank

Tank for separated water

Tank for light oil

Tank for watery oil

Tank for heavy oil

To the deep cooling installation

10. Diagram of Deep Cooling Installation Page 1148 of text

Agitator

Evaporator of sols

Vapor

Gas blower

From condensation installation

To the furnaces

Centrifugal pump

NH_3 evaporator

Symbols TK4-Deep cooler

CaCl2-Cooling columns

D 1-3-Washing columns

W. A-Heat exchanger

T- Topper

K 1-2-Water cooler

F- Salt filter

Indicator

Continuously running

Controllable

Intermittently running

Pumps

- - - - Gas

———— CaCl-solution

—— . —— Gasoline wash

—— . . . —— Gas with gasoline content

The deep cooler is cooled directly by NH_3

11. Inside View of Deep Cooling Installation Page 1149 of text